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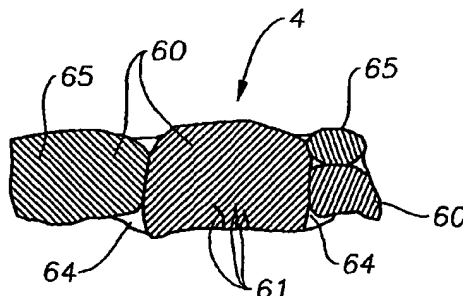
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(54) Title: POLYCRYSTALLINE MATERIAL ELEMENT WITH IMPROVED WEAR RESISTANCE AND METHODS OF MANUFACTURE THEREOF



(57) Abstract: The present invention provides a superhard polycrystalline diamond or diamond-like element with improved wear resistance. Collectively called PCD elements for the purposes of this specification, these elements are formed with a binder-catalyzing material in a high-temperature, high-pressure (HTHP) process. The diamond material is formed and integrally bonded to a substrate containing the catalyzing material during the HTHP process. The diamond body so formed has a working surface, a plurality of crystal being exposed at the working surface, and wherein the exposed crystals are substantially free of microfractures. The exposed parts of the exposed crystals are of rounded or domed form.

## **Polycrystalline Material Element with Improved Wear Resistance**

### **And Methods of Manufacture Thereof**

#### **BACKGROUND OF THE INVENTION**

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##### **1. Field of the Invention.**

The invention relates to superhard polycrystalline material elements for wear, cutting, drawing, and other applications where engineered superhard  
10 surfaces are needed. The invention particularly relates to polycrystalline diamond and polycrystalline diamond-like (collectively called PCD) elements with greatly improved wear resistance and methods of manufacturing them.

##### **2. Description of Related Art.**

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Polycrystalline diamond and polycrystalline diamond-like elements are known, for the purposes of this specification, as PCD elements. PCD elements are formed from carbon based materials with exceptionally short inter-atomic distances between neighboring atoms. One type of diamond-like material  
20 similar to PCD is known as carbonitride (CN) described in U.S Patent No. 5,776,615. In general, PCD elements are formed from a mix of materials

processed under high-temperature and high-pressure into a polycrystalline matrix of inter-bonded superhard carbon based crystals. A common trait of PCD elements is the use of catalyzing materials during their formation, the residue from which often imposes a limit upon the maximum useful operating  
5 temperature of the element while in service.

A well known, manufactured form of PCD element is a two-layer or multi-layer PCD element where a facing table of polycrystalline diamond is integrally bonded to a substrate of less hard material, such as tungsten carbide. The PCD  
10 element may be in the form of a circular or part-circular tablet, or may be formed into other shapes, suitable for applications such as hollow dies, friction bearings, valve surfaces, indentors, tool mandrels, etc. PCD elements of this type may be used in almost any application where a hard wear and erosion resistant material is required. The substrate of the PCD element may be brazed to a carrier, often  
15 also of cemented tungsten carbide. This is a common configuration for PCDs used as cutting elements, for example in fixed cutter or rolling cutter earth boring bits when received in a socket of the drill bit, or when fixed to a post in a machine tool for machining. These PCD elements are typically called polycrystalline diamond cutters (PDC).

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There are numerous variations in the methods of manufacture of these PDC elements. For example various ranges of average diamond particle sizes may be utilized in the manufacture to enhance wear properties as shown in U.S. Patents Nos, 4,861,350; 5,468,268; and 5,545,748 all herein incorporated by reference  
5 for all they disclose. Also, methods to provide a range of wear resistance across or into the working surface of a PDC are shown in U.S. Patent Nos. 5,135,061 and 5,607,024 also herein incorporated by reference for all they disclose. However, because the wear resistance is varied by changing the average size of the diamond particles, there is an inherent trade-off between impact strength and  
10 wear resistance in these designs. As a consequence, the PDC elements with the higher wear resistance will tend to have poor impact strength, which for PDC's used in drilling applications, is often unacceptable.

Typically, higher diamond volume densities in the diamond table increases  
15 wear resistance at the expense of impact strength. However, modern PDC elements typically utilize often complex geometrical interfaces between the diamond table and the substrate as well as other physical design configurations to improve the impact strength. Although this allows wear resistance and impact strength to be simultaneously maximized, the tradeoff still exists, and has not  
20 significantly changed for the past several years prior to the present invention.

PCD elements are most often formed by sintering diamond powder with a suitable binder-catalyzing material in a high-pressure, high-temperature press. One particular method of forming this polycrystalline diamond is disclosed in U.S. Patent No. 3,141,746 herein incorporated by reference for all it discloses. In one common process for manufacturing PCD elements, diamond powder is applied to the surface of a preformed tungsten carbide substrate incorporating cobalt. The assembly is then subjected to very high temperature and pressure in a press. During this process, cobalt migrates from the substrate into the diamond layer and acts as a binder-catalyzing material, causing the diamond particles to bond to one another with diamond-to-diamond bonding, and also causing the diamond layer to bond to the substrate.

After completion of the sintering process, a finishing operation is performed which typically includes a step of machining the PCD. The machining operation may be similar to that described in US Patent No 5,447,208 in which progressively smaller diamond grit particles are used to produce a highly polished working surface on the diamond material. However, the use of progressively small grit sizes is not essential, and depending upon the application in which the PCD is to be used, a less polished finish may be

acceptable, in which case the machining operation may be performed with, say, only one relatively coarse diamond grit size.

Regardless as to the finish to be produced, the action of the diamond grit on  
5 the working surface of the PCD during the machining operation is to cause slightly projecting parts of the PCD (at a microscopic level) to be worn or broken away from the remainder thereof to flat a working surface of good flatness. Where the machining operation is used to produce a highly polished finish, then the finish working surface is of very good flatness, but will still  
10 include small scratches formed by the action of the diamond grit on the working surface. In addition to forming a surface of good flatness, the machining operation causes the formation of very small fractures, referred to herein as microfractures, in the diamond crystals which are exposed at the working surface. These crystals may also be subject to other damage during the  
15 machining operation. It is thought that, in subsequent use, cracks may propagate relatively easily along the microfractures, and hence that the PCD finished in this manner is relatively weak, being of relatively low wear resistance.

Although, as described hereinbefore, cobalt is most commonly used as the  
20 binder-catalyzing material, any group VIII element, including cobalt, nickel, iron, and alloys thereof, may be employed.

In an alternative form of thermally stable polycrystalline diamond, silicon is used as the catalyzing material. The process for making polycrystalline diamond with a silicon catalyzing material is quite similar to that described above, except  
5 that at synthesis temperatures and pressures, most of the silicon is reacted to form silicon carbide, which is not an effective catalyzing material. The thermal resistance is somewhat improved, but thermal degradation still occurs due to some residual silicon remaining, generally uniformly distributed in the interstices of the interstitial matrix. There are mounting problems with this type  
10 of PCD element because there is no bondable surface.

More recently, a further type of PCD has become available in which carbonates, such as powdery carbonates of Mg, Ca, Sr, and Ba are used as the binder-catalyzing material when sintering the diamond powder. PCD of this  
15 type typically has greater wear-resistance and hardness than the previous types of PCD elements. However, the material is difficult to produce on a commercial scale since much higher pressures are required for sintering than is the case with conventional and thermally stable polycrystalline diamond. One result of this is that the bodies of polycrystalline diamond produced by this method are smaller  
20 than conventional polycrystalline diamond elements. Again, thermal degradation may still occur due to the residual binder-catalyzing material remaining in the

interstices. Again, because there is no integral substrate or other bondable surface, there are difficulties in mounting this material to a working surface.

### **BRIEF SUMMARY OF THE INVENTION**

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The present invention provides a superhard polycrystalline diamond or diamond-like element with improved wear resistance. Collectively called PCD elements for the purposes of this specification, these elements are formed with a binder-catalyzing material in a high-temperature, high-pressure (HTHP) process.

10

The diamond material is formed and integrally bonded to a substrate containing the catalyzing material during the HTHP process. The diamond body so formed has a working surface, a plurality of crystals being exposed at the working surface, and wherein the crystals exposed at at least a portion of the working surface are substantially free of microfractures.

15

It will be appreciated that by providing a PCD element in which at least some of the crystals at the working surface are treated to be substantially free of microfractures, the likelihood of cracks propagating through the crystals is reduced, and as a consequence, the wear resistance of the element is improved and the useful working life of the PCD is increased.

20



The PCD element may be used in a wide range of applications. By way of example only, the PCD element may find applications in downhole equipment, either in cutters or for use in wear resistant bearings. The PCD element may alternatively be used in hollow dies, friction bearings, valve surfaces, indentors, tool mandrels and a wide range of other applications. Additionally, the PCD element may be used in the machining of abrasive materials, for example abrasive wood materials, ferrous and non-ferrous materials, and hard or very abrasive engineering materials such as stone, asphalt and the like.

In accordance with one aspect of the present invention there is provided a PCD element comprising a body including matrix of crystals of a superhard material, the body having a surface, a plurality of the crystals being exposed at the surface, wherein at least the exposed part of each of the crystals exposed at at least a portion of the surface is of rounded form. Crystals of this form are substantially free of microfractures and other defects and so a PCD of this form is of improved wear resistance.

The PCD element conveniently comprises a substrate having a front surface, a table of superhard material being bonded to the substrate, the table of superhard material comprising a matrix of crystals. The crystals of superhard material conveniently comprise diamond crystals.

The invention also relates to a PCD element which comprises a substrate having a front surface, a table of superhard material being bonded to the front surface of the substrate, the table of superhard material comprising a matrix of crystals, the table having a surface at which a plurality of the crystals are exposed, at least the exposed parts of the crystals which are exposed at at least a portion of the surface being substantially free of microfractures.

The exposed parts of the exposed crystals are preferably of rounded form.

10

In accordance with another aspect of the invention there is provided a method of manufacturing a PCD element comprising:

sintering diamond powder with a binder-catalyst material in a high pressure, high temperature press to form a body including a matrix of diamond crystals;

15 performing a machining operation on the body to form a working surface thereon at which a plurality of diamond crystals are exposed; and

treating the body to render the crystals which are exposed at at least a portion of the working surface substantially free of microfractures.

20 The step of treating the body may include performing a machining operation on the body in the absence of a coolant material. Alternatively, the

step of treating the body may include performing a machining operation on the body with a restricted coolant supply. In either case, the temperature of the diamond crystals at the exposed surface is raised, and extremely high local temperatures and pressures are reached such that the exposed diamond surface becomes plastic. Under these conditions, the exposed surface of the diamond crystals can undergo limited plastic flow and thereby deform to generate a smooth rounded surface which is substantially free of microfractures. It is believed that the extreme localized surface temperature attained during this treatment is in excess of 2000°C. It is well known in the art that diamond crystals deform in a plastic manner when subjected to high pressures and temperatures such as during the HTHP process described briefly hereinbefore. The deformation is normally revealed by the presence of 'deformation twinning' within the crystals such as occurs in ductile metals at much lower temperatures. Prior to this invention, this plastic deformation was not known to occur locally at a surface as a result of a machining process, and such machining conditions are normally avoided.

The machining operation may be an ultra-high speed grinding operation.

Alternatively, the step of treating the body may include performing a thermochemical treatment on the body. The thermochemical treatment may

involve the treatment of the working surface of the body with at least one oxidizing compound, or the treatment thereof with at least one iron group element.

5 In accordance with another aspect of the invention there is provided a method of manufacturing a PCD element comprising:

sintering diamond powder with a binder-catalyst material in a high pressure, high temperature press to form a body including a matrix of diamond crystals;

performing a machining operation on the body to form a working surface  
10 thereon at which a plurality of diamond crystals are exposed; and

treating the body to render at least the exposed parts of the crystals exposed at at least a portion of the working surface of rounded form.

Crystals treated so as to be of rounded form are substantially free of  
15 microfractures and other surface defects, and so a PCD manufactured in accordance with the invention has the advantage that it is of improved wear resistance as the likelihood of cracks propagating through the crystals thereof is reduced.

20

#### BRIEF DESCRIPTION OF THE DRAWINGS

The invention will further be described, by way of example only, with reference to the accompanying drawings, in which:

5        Figure 1 is a typical PCD element of the present invention.

Figure 2 is a micro-structural representation of part of the PCD element.

Figure 3 is a diagrammatic representation illustrating the crystal structure of  
10      part of a typical PCD element close to a working surface thereof.

Figure 4 is a view similar to Figure 3 but illustrating the structure of a PCD  
in accordance with an embodiment of the invention.

15        Figure 5 is a typical PCD of the present invention shown as a cutting  
element.

Figure 6 is a perspective view of an insert used in machine tools utilizing  
the PCD element of the present invention.

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Figure 7 is a perspective view of a dome shaped PCD element suitable for use in both rolling cutter drill bits and in fixed cutter drill bits.

Figure 8 is a side view of a fixed cutter rotary drill bit using a PCD element  
5 of the present invention.

Figure 9 is a perspective view of a rolling cutter rotary drill bit using a PCD element of the present invention.

Figure 10 is a section view of a wire drawing die having a PCD element of  
10 the present invention.

Figure 11 is perspective view of a bearing having a PCD element of the present invention.

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Figures 12 and 13 are front views of the mating parts of a valve having a PCD element of the present invention.

Figure 14 is a side view of an indenter having a PCD element of the present  
20 invention.

Figure 15 is a partial section view of a punch having a PCD element of the present invention.

Figure 16 is perspective view of a measuring device having a PCD element  
5 of the present invention.

### **DETAILED DESCRIPTION OF THE INVENTION AND THE PREFERRED EMBODIMENT**

10 The polycrystalline diamond or diamond-like material (PCD) element 2 of the present invention is shown in Figure 1. The PCD element 2 has a plurality of partially bonded superhard, diamond or diamond-like, crystals 60, (shown in Figure 2) a catalyzing material 64, and an interstitial matrix 68 formed by the interstices 62 among the crystals 60. The element 2 also has one or more  
15 working surfaces 4 and the diamond crystals 60 and the interstices 62 form the volume of the body 8 of the PCD element 2. Preferably, the element 2 is integrally formed with a metallic substrate 6, typically tungsten carbide with a cobalt binder material. To be effective when used in an abrasive wear application, the volume density of the diamond in the body 8 should be greater  
20 than 85 volume %, and preferably be higher than 90%.

The working surface 4 is any portion of the PCD body 8 which, in operation, may contact the object to be worked. In this specification, when the working surface 4 is discussed, it is understood that it applies to any portion of the body 8 which may be exposed and/or used as a working surface.

5 Furthermore, any portion of any of the working surface 4 is, in and of itself, a working surface.

During manufacture, under conditions of high-temperature and high-pressure (HTHP), the interstices 62 among the crystals 60 fill with the catalyzing

10 material 64 followed by bonds forming among the crystals 60.

Referring now to Figure 2, it is well known that there is a random crystallographic orientation of the diamond or diamond-like crystals 60 as shown by the parallel lines 61 representing the cleavage planes of each crystal 60. As

15 can be seen, adjacent crystals 60 have bonded together with interstitial spaces 62 among them. Because the cleavage planes 61 are oriented in different directions on adjacent crystals 60 there is generally no straight path available for diamond fracture. This structure allows PCD materials to perform well in extreme loading environments where high impact loads are common.



In the process of bonding the crystals 60 in a high-temperature, high-pressure press, the interstitial spaces 62 among the crystals 60 become filled with a binder-catalyzing material 64. It is this catalyzing material 64 that allows the bonds to be formed between adjacent diamond crystals 60 at the relatively  
5 low pressures and temperatures present in the press.

The average diamond volume density in the body 8 of the PCD element 2 of the present invention ranges from about 85% to about 99%. The high diamond volume density is achieved by using diamond crystals 60 with a range of particle  
10 sizes, with an average particle size ranging from about 30 to about 60 microns. Typically, the diamond mixture may comprise 20% to 60% diamond crystals 60 in the 5-15 micron range, 20% to 40% diamond crystals 60 in the 25-40 micron range, and 20% to 40% diamond crystals 60 in the 50-80 micron diameter range, although numerous other size ranges and percentages may be used. This mixture  
15 of large and small diamond crystals 60 allows the diamond crystals 60 to have relatively high percentages of their outer surface areas dedicated to diamond-to-diamond bonding, often approaching 95%, contributing to a relatively high apparent abrasion resistance.

20 After completion of the sintering operation to form the body 8 of the PCD element 2, and to bond the element 2 to the substrate 6, a cleaning operation in

performed on the element 2. The cleaning operation includes a finishing operation of machining the working surfaces 4 of the element 2 to improve the smoothness thereof and to remove macroscopic defects therefrom. The crystals 60 of the element 2 include a plurality of crystals 65 exposed at the working surfaces 4. As described hereinbefore, it is thought that the machining operation induces the formation of microfractures 63 and other defects into the crystals 65 of the element 2 which are exposed at the working surface 4, as shown in Figure 3. The formation of these defects in the crystals 65 which are exposed at the working surface 4 may result in the element 2 being weakened as, in use, cracks may propagate through the crystals 65, starting at the microfractures 63, and then typically following the cleavage planes 61 of the crystals.

In accordance with the invention, rather than simply performing a machining operation on the element 2, the element 2 is treated to render the crystals 65 exposed at the working surfaces 4 thereof, or at at least a portion of the working surface thereof, for example around at least a part of an edge portion 5 of the element 2, substantially free of microfractures 70 and other surface defects. The treatment comprises performing a machining operation, for example an ultra-high speed grinding operation, in the absence of a coolant, or with a restricted coolant supply so that the machining operation is performed at a significantly higher surface temperature than usual. By way of example, the

surface temperature reached may be in excess of 2000°C. The performance of the machining operation under such high temperature conditions is thought to affect the way in which material is removed from the element 2 such that the removal of the material does not result in the formation of microfractures or other defects in the exposed parts of the exposed crystals 65. Rather, the exposed diamond surface undergoes limited plastic deformation, the diamond flowing to form a rounded surface which is substantially free of microfractures. Figure 4 is a diagrammatic view similar to Figure 3 illustrating the effect of performing such a machining operation. As shown in Figure 4, the exposed parts of the crystals 65 exposed at the working surfaces 4 are of rounded or domed form, and are substantially free of stress raisers in the form of microfractures, cracks or other surface defects. As a result, in use, the risk of cracks forming in the crystals 65 is reduced, and hence the wear resistance of the element 2 is improved.

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Although as described above the step of treating the element 2 to render its working surfaces 4 substantially free of microfractures and other surface defects may involve performing a machining operation in the absence of coolant or with a restricted supply of coolant, it will be appreciated that the element 2 could be treated in other ways to achieve the same result. For example, a thermochemical treatment operation could be performed on the element 2 using, for example,

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iron group elements or oxidizing compounds. Further, combinations of these techniques may be used.

As indicated hereinbefore, a PCD element so manufactured may find application in a wide range of uses. One particularly useful application for the PCD element 2 of the present invention is as cutting elements 10, 50, 52 as shown in Figures 5, 6 and 7. The working surface of the PCD cutting elements 10, 50, 52 may be a top working surface 70 and/or a peripheral working surface 72. The PCD cutting element 10 of Figure 5 is one that may be typically used in fixed cutter type rotary drill bits 12, or for gauge protection in other types of downhole tools. The PCD cutting element 50 shown in Figure 7 may be shaped as a dome 39. This type of PCD cutting element 50 has an extended base 51 for insertion into sockets in a rolling cutter drill bit 38 or in the body of both types of rotary drill bits, 12, 38 as will be described in detail.

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The PCD cutting element 52 of Figure 6 is adapted for use in a machining process. Although the configuration of the cutting element 52 in Figure 6 is rectangular, it would be appreciated by those skilled in the art that this element could be triangular, quadrilateral or many other shapes suitable for machining highly abrasive products that are difficult to machine with conventional tools.

20

The PCD cutting element 10 may be a preform cutting element 10 of a fixed cutter rotary drill bit 12 (as shown in Figure 8). The bit body 14 of the drill bit is formed with a plurality of blades 16 extending generally outwardly away from the central longitudinal axis of rotation 18 of the drill bit. Spaced apart side-by-side along the leading face 20 of each blade is a plurality of the PCD cutting elements 10 of the present invention.

Typically, the PCD cutting element 10 has a body in the form of a circular tablet having a thin front facing table 30 of diamond or diamond-like (PCD) material, bonded in a high-pressure high-temperature press to a substrate 32 of less hard material such as cemented tungsten carbide or other metallic material. The cutting element 10 is preformed and then typically bonded on a generally cylindrical carrier 34 which is also formed from cemented tungsten carbide, or may alternatively be attached directly to the blade. The PCD cutting element 10 has working surfaces 70 and 72.

The cylindrical carrier 34 is received within a correspondingly shaped socket or recess in the blade 16. The carrier 34 will usually be brazed or shrink fit in the socket. In operation the fixed cutter drill bit 12 is rotated and weight is applied. This forces the cutting elements 10 into the earth being drilled, effecting a cutting and/or drilling action.

The PCD cutting elements 10 may also be applied to the gauge region 36 of the bit 12 to provide a gauge reaming action as well as protecting the bit 12 from excessive wear in the gauge region 36. In order to space these cutting elements 10 as closely as possible, it may be desirable to cut the elements into shapes, such as the rectangular shape shown, which more readily fit into the gauge region 36.

In a second embodiment, the cutting element 50 (as shown in Figure 7) of the present invention is on a rolling cutter type drill bit 38, shown in Figure 9. A rolling cutter drill bit 38 typically has one or more truncated rolling cone cutters 40, 41, 42 assembled on a bearing spindle on the leg 44 of the bit body 46. The cutting elements 50 may be mounted as one or more of a plurality of cutting inserts arranged in rows on rolling cutters 40, 41, 42, or alternatively the PCD cutting elements 50 may be arranged along the leg 44 of the bit 38. The PCD cutting element 50 has a body in the form of a facing table 35 of diamond or diamond like material bonded to a less hard substrate 37. The facing table 35 in this embodiment of the present invention is in the form of a domed surface 39 and has working surfaces 70 and 72. Accordingly, there are often a number of transitional layers between the facing table 35 and the substrate 37 to help more

evenly distribute the stresses generated during fabrication, as is well known to those skilled in the art.

In operation the rolling cutter drill bit 38 is rotated and weight is applied.  
5 This forces the cutting inserts 50 in the rows of the rolling cone cutters 40, 41, 42 into the earth, and as the bit 36 is rotated the rolling cutters 40, 41, 42 turn, effecting a drilling action.

In another embodiment, the PCD cutting element 52 of the present  
10 invention is in the form of a triangular, rectangular or other shaped material for use as a cutting insert in machining operations. In this embodiment, the cutting element 52 has a body in the form of a facing table 54 of diamond or diamond like material bonded to a less hard substrate 56 with working surfaces 70 and 72. Typically, the cutting element 52 would then be cut into a plurality of smaller  
15 pieces which are subsequently attached to an insert 58 that is mounted in the tool holder of a machine tool. The cutting element 52 may be attached to the insert by brazing, adhesives, welding, or clamping. It is also possible to finish form the cutting element 52 in the shape of the insert in a high-temperature high-pressure manufacturing process.

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As shown in Figures 10-16, PCD elements 2, 102, 202 of the present invention may also be used for other applications such as hollow dies, shown for example as a wire drawing die, 300 of Figure 10 utilizing a PCD element 302 of the present invention.

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Other applications include friction bearings 320 with a PCD bearing element 322 shown in Figure 11 and the mating parts of a valve 340, 344 with surfaces 342 having a PCD element 342 of the present invention as shown in Figures 12 and 13. In addition, indentors 360 for scribes, hardness testers, surface roughening, etc. may have PCD elements 362 of the present invention as shown in Figure 14. Punches 370 may have either or both dies 372, 374 made of the PCD material of the present invention, as shown in Figure 15. Also, tool mandrels 382 and other types of wear elements for measuring devices 380, shown in Figure 16 may be made of PCD elements of the present inventions.

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Whereas the present invention has been described in particular relation to the drawings attached hereto, it should be understood that other and further modifications apart from those shown or suggested herein, may be made within the scope and spirit of the present invention.

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## CLAIMS

## WHAT IS CLAIMED IS:

1. A PCD element comprising a body including a matrix of crystals of a  
5 superhard material, the body having a surface, a plurality of the crystals of the  
matrix being exposed at the surface, wherein at least the exposed part of each of  
the crystals exposed at at least a portion of the surface is of rounded form.
2. A PCD element according to Claim 1, further comprising a substrate to  
10 which the body is bonded.
3. A PCD element according to Claim 1, wherein the matrix of crystals defines  
a plurality of interstices, at least some of the interstices containing a binder-  
catalyst material.  
15
4. A PCD element according to Claim 3, wherein the binder-catalyst material  
comprises a Group VIII material.
5. A PCD element according to Claim 4, wherein the binder-catalyst  
20 comprises cobalt.

6. A PCD element according to Claim 1, wherein the crystals of superhard material comprise diamond crystals.

7. A PCD element comprising a substrate having a front face, a table of superhard material being bonded to the front face of the substrate, the table of superhard material comprising a matrix of crystals, the table having a surface at which a plurality of the crystals are exposed, at least the exposed parts of the crystals exposed at at least a portion of the surface being substantially free of microfractures.

8. A PCD element according to Claim 7, wherein the exposed parts of the exposed crystals are of rounded form.

9. A PCD element according to Claim 7, wherein the matrix of crystals defines a plurality of interstices, at least some of the interstices containing a binder-catalyst material.

10. A PCD element according to Claim 9, wherein the binder-catalyst material comprises a Group VIII material.

11. A PCD element according to Claim 10, wherein the binder-catalyst comprises cobalt.

12. A PCD element according to Claim 7, wherein the crystals of superhard  
5 material comprise diamond crystals.

13. A method of manufacturing a PCD element comprising:  
sintering diamond powder with a binder-catalyst material in a high pressure,  
high temperature press to form a body including a matrix of diamond crystals;  
10 performing a machining operation on the body to form a working surface  
thereon at which a plurality of diamond crystals are exposed; and  
treating the body to render the crystals which are exposed at at least a  
portion of the working surface substantially free of microfractures.

14. A method according to Claim 13, wherein the binder-catalyst material is a  
15 Group VIII material.

15. A method according to Claim 14, wherein the binder-catalyst material is  
cobalt.

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16. A method according to Claim 13, further comprising sintering the diamond powder with a substrate in the high pressure, high temperature press to bond the body to the substrate.

5 17. A method according to Claim 16, wherein the substrate is a tungsten carbide substrate.

18. A method according to Claim 13, wherein the step of treating the body includes performing a machining operation on the body in the absence of a  
10 coolant material.

19. A method according to Claim 13, wherein the step of treating the body includes performing a machining operation on the body with a restricted coolant supply.

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20. A method according to Claim 13, wherein the step of treating the body includes performing a thermochemical treatment on the body.

21. A method according to Claim 20, wherein the thermochemical treatment  
20 involves the treatment of the working surface of the body with at least one oxidizing compound.

22. A method according to Claim 20, wherein the thermochemical treatment involves the treatment of the working surface of the body with at least one iron group element.

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23. A method according to Claim 13, wherein a local temperature in excess of 2000°C is reached at at least a portion of the working surface during the step of treating the body.

10

24. A method of manufacturing a PCD element comprising:

sintering diamond powder with a binder-catalyst material in a high pressure, high temperature press to form a body including a matrix of diamond crystals;

performing a machining operation on the body to form a working surface thereon at which a plurality of diamond crystals are exposed; and

15 treating the body to render at least the exposed parts of the crystals exposed at at least a portion of the working surface of rounded form.

25. A method according to Claim 24, wherein the binder-catalyst material is a Group VIII material.

20

26. A method according to Claim 25, wherein the binder-catalyst material is cobalt.

27. A method according to Claim 24, further comprising sintering the diamond  
5 powder with a substrate in the high pressure, high temperature press to bond the body to the substrate.

28. A method according to Claim 27, wherein the substrate is a tungsten carbide substrate.

10

29. A method according to Claim 24, wherein the step of treating the body includes performing a machining operation on the body in the absence of a coolant material.

15 30. A method according to Claim 24, wherein the step of treating the body includes performing a machining operation on the body with a restricted coolant supply.

31. A method according to Claim 24, wherein the step of treating the body  
20 includes performing a thermochemical treatment on the body.

32. A method according to Claim 31, wherein the thermochemical treatment involves the treatment of the working surface of the body with at least one oxidizing compound.

5 33. A method according to Claim 31, wherein the thermochemical treatment involves the treatment of the working surface of the body with at least one iron group element.

34. A method according to Claim 25, wherein a local temperature in excess of  
10 2000°C is reached at at least a portion of the working surface during the step of treating the body.

35. A PCD element according to Claim 1, comprising a preform cutting element having a facing table and a cutting surface, wherein the cutting element is  
15 mounted upon a cutting face of a fixed cutter rotary drill bit.

36. A PCD element according to Claim 1, comprising a preform cutting element having a facing table and a cutting surface, wherein the cutting element is mounted upon a body of a rolling cutter drill bit.

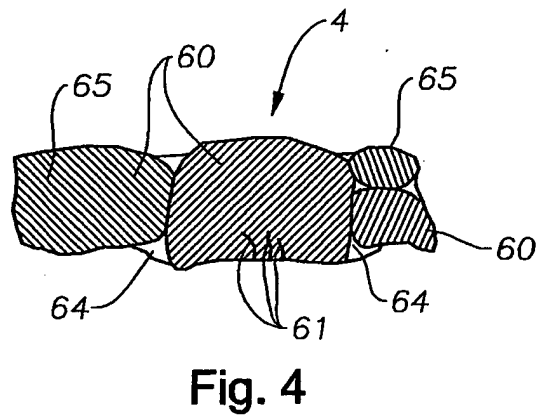
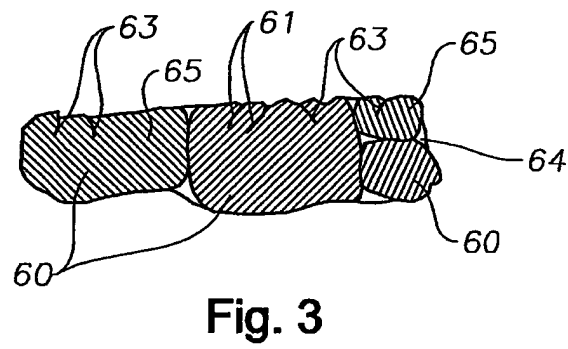
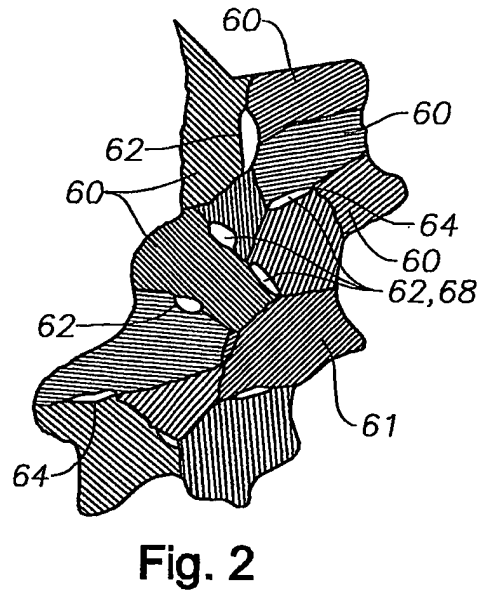
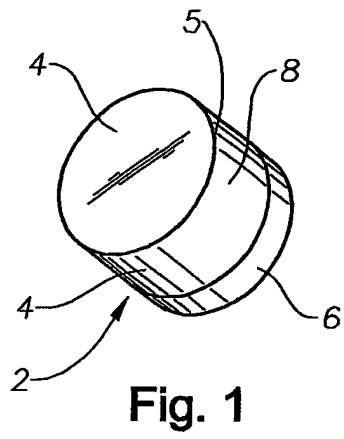
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37. A PCD element according to Claim 1, comprising a cutting element with a cutting surface adapted for use as a cutting insert in a machining operation.

38. A PCD element according to Claim 1, comprising a drawing die.

5





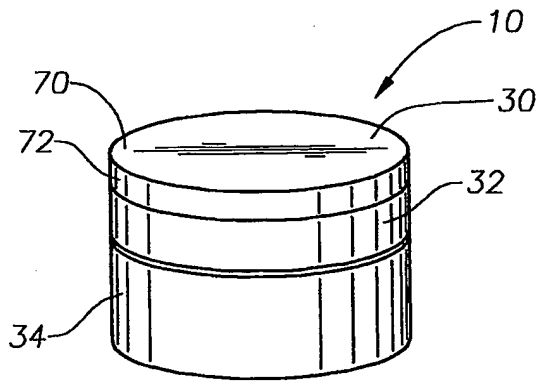


Fig. 5

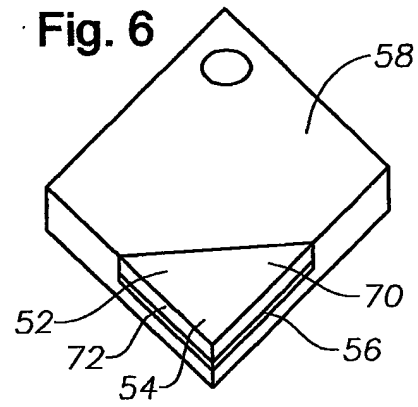


Fig. 6

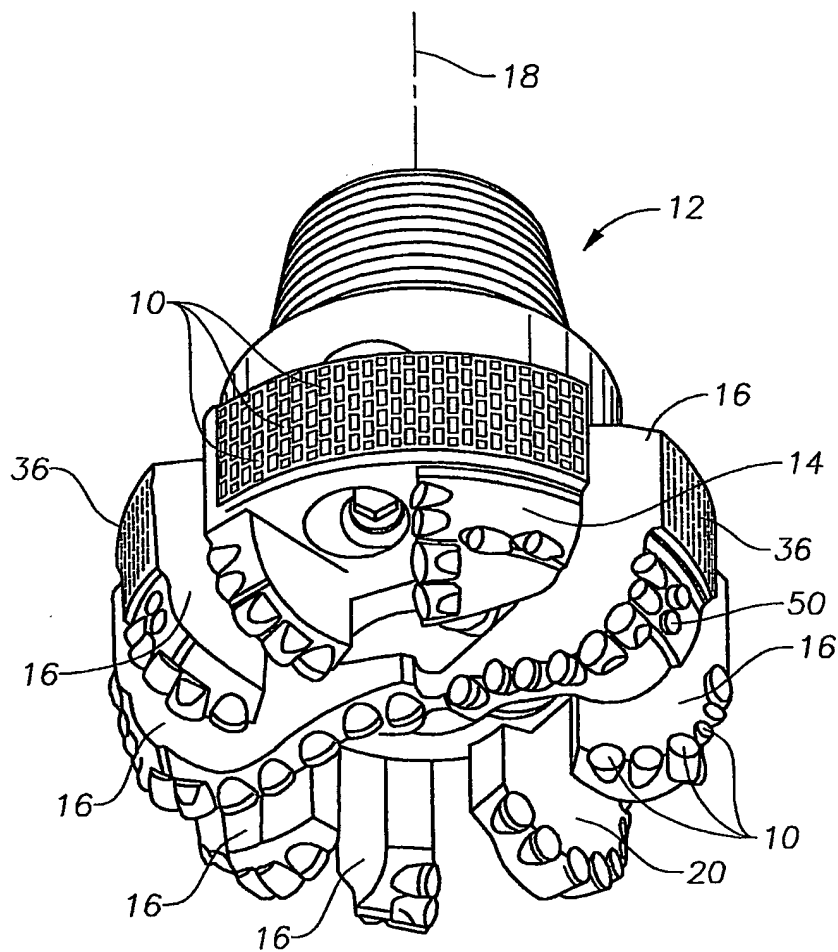
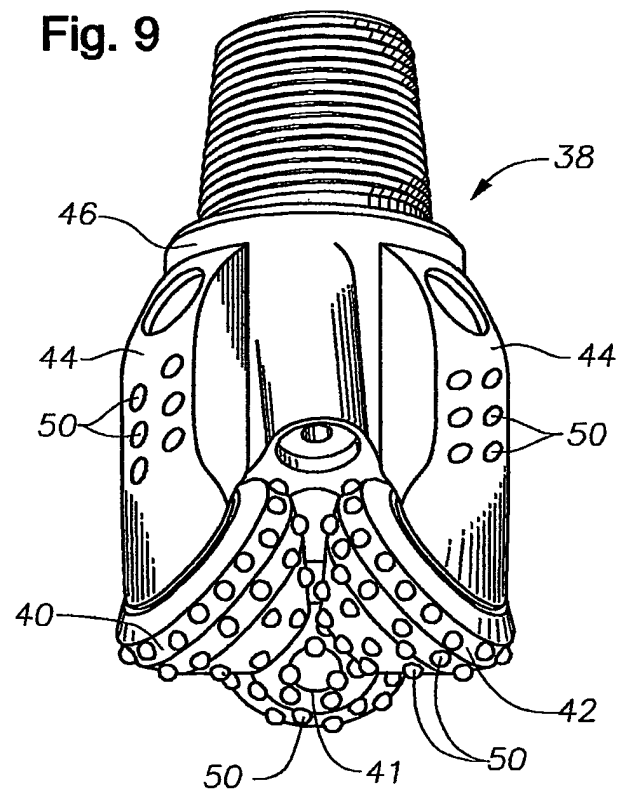
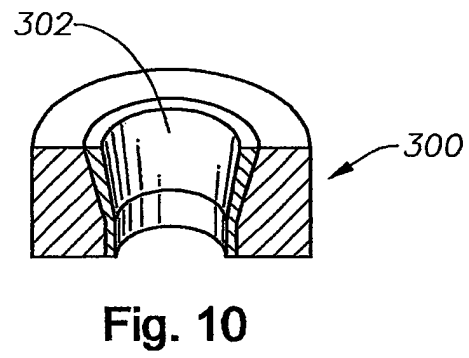
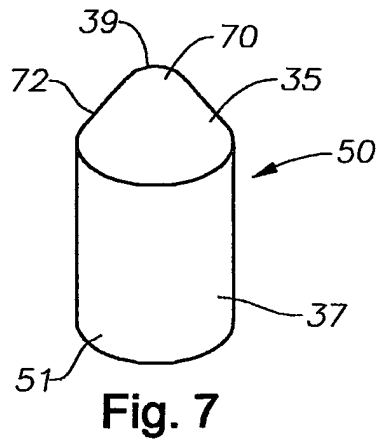
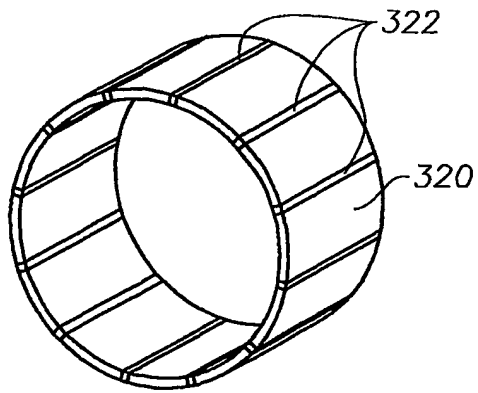
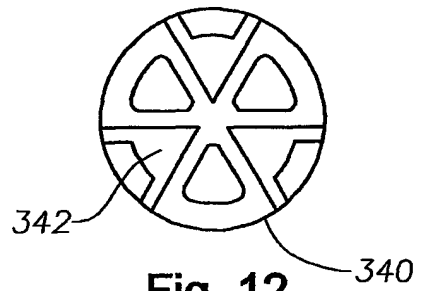


Fig. 8

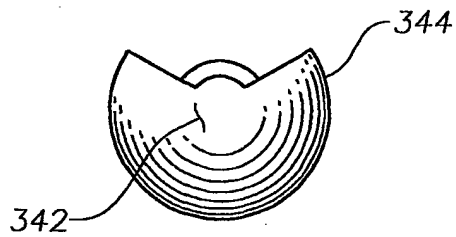




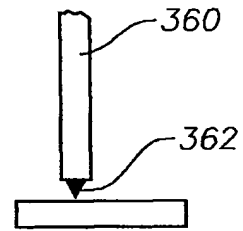
**Fig. 11**



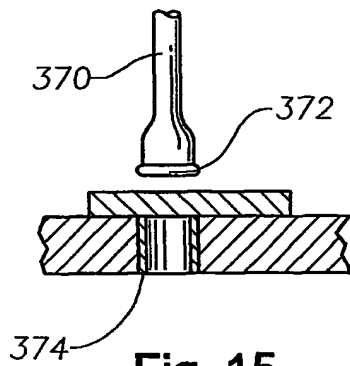
**Fig. 12**



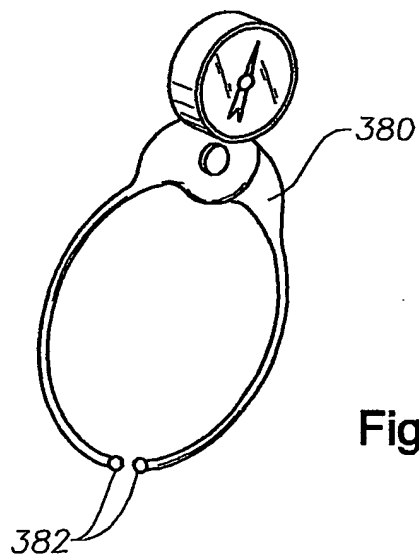
**Fig. 13**



**Fig. 14**



**Fig. 15**



**Fig. 16**

# INTERNATIONAL SEARCH REPORT

Internat Application No  
PCT/GB 03/00642

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 C22C26/00 C04B35/52

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C22C C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

COMPENDEX, INSPEC, EPO-Internal, CHEM ABS Data, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	LARSSON P ET AL: "Wear of a new type of diamond composite" INT J REFRACT MET HARD MATER; INTERNATIONAL JOURNAL OF REFRACTORY METALS AND HARD MATERIALS 1999 ELSEVIER APPLIED SCIENCE PUBL LTD, EXETER, ENGL, vol. 17, no. 6, 1999, pages 453-460, XP002239906 page 456  --- -/-	1-33, 35-38

☒ Further documents are listed in the continuation of box C.

☐ Patent family members are listed in annex.

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Date of the actual completion of the international search

28 May 2003

Date of mailing of the international search report

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## INTERNATIONAL SEARCH REPORT

Internal Application No

PCT/GB 03/00642

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>SUMIYA H ET AL: "Synthesis of polycrystalline diamond with new non-metallic catalyst under high pressure and high temperature" INTERNATIONAL JOURNAL OF REFRACTORY METALS &amp; HARD MATERIALS, 1999, ELSEVIER, UK, vol. 17, no. 5, pages 345-350, XP002239907 ISSN: 0263-4368 page 348 -page 349</p>	<p>1-12, 35-38</p>